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Test Procedure for Iodine-131 in Drinking Water: Interlaboratory Collaborative Study

Monsanto Research Corp., Miamisburg, OH

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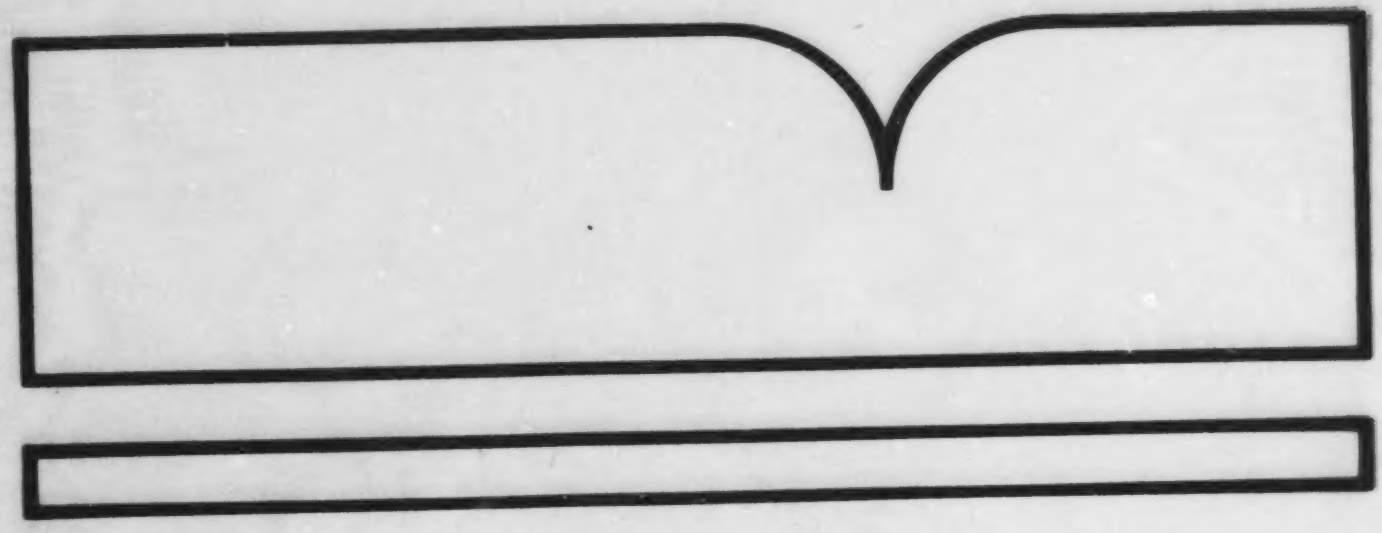
May 83



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TEST PROCEDURE FOR IODINE-131 IN DRINKING WATER: Interlaboratory Collaborative Study

by

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16. ABSTRACT

An interlaboratory collaborative study was conducted to test a procedure for the measurement of iodine-131 in drinking water. Drinking water samples, containing 7.8, 25.9, and 78.3 pCi/l, were analyzed in triplicate. Of the 15 participants, data from four were rejected, and a statistical evaluation on the data from 11 participants was done.

The statistical analyses of the test results gave coefficients of variation for repeatability (within-laboratory precision) of 8.7, 10.5, and 7.7 percent for the three samples for an average repeatability precision of 9.0 percent over the iodine-131 concentration range of 8 to 80 pCi/l. The analyses also gave coefficients of variation for reproducibility (combined within- and between-laboratory precision) of 17.4, 15.5, and 15.7 percent for an average reproducibility precision of 16.2 percent over the concentration range of 8 to 80 pCi/l.

The accuracy indexes of the test procedure for the three iodine-131 concentrations was 97.4, 97.3, and 84.8 percent for an average of 93.2 percent over the concentration range of 8 to 80 pCi/l.

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ABSTRACT

A selected test procedure for measuring the concentration of iodine-131 in drinking water samples was tested by an interlaboratory collaborative study. The purpose of the study was to determine an estimated precision and accuracy for the test procedure that can be expected in the use of the procedure by any competent laboratory to analyze drinking water samples for iodine-131.

Drinking water samples containing iodine-131 at concentrations of 7.8, 25.9, and 78.3 pCi/l (on the designated collection date) were analyzed in triplicate by 11 collaborators (10 laboratories) using the test procedure.

A statistical analysis of the test results gave coefficients of variation for repeatability (within-laboratory precision) of 8.7, 10.5, and 7.7 percent respectively for the three samples for an average repeatability precision of 9.0 percent. The analysis also gave coefficients of variation for reproducibility (combined within- and between-laboratory precision) of 17.4, 15.9 and 15.7 percent for the respective samples for an average reproducibility precision of 16.2 percent.

Test results of the study showed average iodine recoveries of 76.4, 79.3, and 79.8 percent respectively for the three samples for an overall average of 78.5 percent.

A comparison of the grand average test results with the known values for the three samples, shows accuracy indexes of 97.4, 97.3, and 84.8 percent respectively, for an average accuracy index of 93.2 for the test method.

The test procedure used in this study was listed in the EPA approved procedures manual, EPA-600/4-75-008 (Revised) March 1976 "Interim Radiochemical Methodology For Drinking Water" and is repeated in the updated version of that manual, EPA-600/4-80-032 August 1980 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water".

This report is submitted as partial fulfillment of an Interagency Agreement, EPA-IAG-79-D-X0736, between the Environmental Protection Agency and the Department of Energy. Work was done by the Mound Facility of the Monsanto Research Corporation under Contract No. DE-AC04-76-DP00053. The work covered the period of September 1, 1979 to June 15, 1980.

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LIST OF ABBREVIATIONS AND SYMBOLS

ABBREVIATIONS

ASTM	-- The American Society for Testing and Materials
Cl^-	-- chloride
c/m	-- counts per minute
d/m	-- disintegrations per minute
DOE	-- The Department of Energy
EPA	-- Environmental Protection Agency
g/cm^3	-- grams per cubic centimeter
g/l	-- gram/liter
IAG	-- Interagency Agreement
kg	-- kilogram
MCL	-- maximum contaminant level
mg/l	-- milligram/liter
nCi/g	-- nanocurie/gram
NIPDWR	-- National Interim Primary Drinking Water Regulations
pCi/l	-- picocuries per liter
ppm	-- parts per million
µCi/g	-- microcurie/gram
2S %	-- Two sigma percent (two standard deviations in percent)

SYMBOLS

$A_j\%$	-- the accuracy index for the sample j level of iodine-131 concentration
d_{ij}	-- the deviation of Lab i average from the grand average for sample j
$\%d_{ij}$	-- the percent deviation of Lab i average from the grand average for sample j
e_{ij}	-- the reduced deviation of Lab i average from the grand average for sample j
I_{r_j}	-- the repeatability interval for sample j level
I_{R_j}	-- the reproducibility interval for sample j level
k_{ij}	-- the ratio of Lab i average standard deviation (S_{ij}) to the pooled standard deviation (S_{r_j}) for sample j
n	-- the number of replicate analyses
P	-- the number of participants in the collaborative study
S_{ij}	-- the standard deviation of the replicate test results for sample j by Lab i
S_{L_j}	-- the standard deviation of between-laboratories precision for sample j

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S_{r_j} -- the repeatability (within-laboratory) standard deviation for the sample j

$\bar{S}_{r_j}$ -- the smoothed repeatability standard deviation for sample j

S_{R_j} -- the reproducibility (combined within- and between-laboratory) standard deviation for sample j

$\bar{S}_{R_j}$ -- the smoothed reproducibility standard deviation for sample j

$S_{\bar{x}_j}$ -- the standard deviation of the grand average for sample j

t_c -- the critical value of the bias test for P participants

t_j -- the bias value for sample j

T_n -- the T-test value of the nth replicate, outlier test

U_j -- the estimated standard error of a cell average, calculated on the basis of replication error only

$V_{L_j}\%$ -- the coefficient of variation for between-laboratories precision for sample j

$V_{r_j}\%$ -- the coefficient of variation for repeatability (within-laboratory) for sample j

$V_{R_j}\%$ -- the coefficient of variation for reproducibility (combined within- and between-laboratory) for sample j

$\bar{X}_h$ -- the test result of the replicate h

$\bar{X}_{ij}$ -- the arithmetic average of all replicate test results of sample j by Lab i

X_{ijh} -- the test result of replicate h of sample j by Lab i

$\bar{X}_j$ -- the grand average value for sample j

Y_j -- the known value of the sample j iodine-131 concentration (pCi/l)

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SECTION 1

INTRODUCTION

The National Interim Primary Drinking Water Regulations (NIPDWR) calls for using approved test procedures for analyzing public drinking water supplies for contaminants. The NIPDWR allows for the use of alternate test procedures that have been approved, based on equivalent precision and accuracy to that of the approved test procedure. This report describes a multilaboratory test of a method selected for the analysis of drinking water samples for iodine-131 concentrations. The purpose of the study was to determine what precision and accuracy could be expected when any competent laboratory uses the method for compliance monitoring of drinking water supplies for iodine-131 contamination.

The NIPDWR requires a sensitivity of measurement (lower detection limit) of one picocurie per liter (1 pCi/l) for the test procedure that is to be used for measuring iodine-131 concentrations in drinking water samples. Iodine-131 decays by emitting a beta particle and a gamma photon. Gamma spectroscopy has good specificity for gamma emitters, but its sensitivity will only meet the required sensitivity (1 pCi/l) if the iodine-131 is first separated to concentrate from multiliter size samples. The method described and tested in this study calls for separating the iodine-131, using carrier iodine, from one or more liters of sample and then counting the beta activity from the separated iodine-131 in a low beta background counting system.

Iodine-131 is a manmade radioactive contaminant. Its health hazard is such that the maximum contaminant level (MCL) allowed in public drinking water supplies is only 3 pCi/l (NIPDWR). Therefore, there is a need for a test procedure with a known or expected precision and accuracy that can be used for compliance monitoring of public drinking water supplies for iodine-131 contamination.

The test procedure used in this study is listed in the EPA approved procedures manual, EPA-600/4-75-008 (Revised) March 1976 and is repeated in the updated version of that manual, EPA-600/4-80-032 August 1980.

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SECTION 2

CONCLUSIONS

The repeatability precision (within-laboratory precision), reproducibility precision (the combined within- and between-laboratory precision), and the accuracy have been determined for the test procedure (Appendix B) by a multilaboratory test in this study, and they become criteria by which to evaluate an alternate test procedure for equivalency.

The estimated repeatability (within-laboratory), single-operator, single machine, same-day, relative precision of the test procedure for the determination of iodine-131 concentrations at the 7.8 pCi/l level (averaged over the 11 participants) is ±1.3 pCi/l (16.7%, 2S%); at the 25.9 pCi/l level (averaged over the 11 participants) is ±5.3 pCi/l (20.5%, 2S%); and at the 78.3 pCi/l level (averaged over the 11 participants) is ±10.3 pCi/l (13.1%, 2S%), for an average 2S percent of 16.8 percent for the range of iodine-131 concentrations of 7.8 to 78.3 pCi/l.

The estimated reproducibility (combined within- and between-laboratory), multioperator (multilaboratory), single-machine, same-day, relative precision of the test procedure in the determination of iodine-131 concentrations at the 7.8 pCi/l level (averaged over the 11 participants) is ±2.6 pCi/l (33.4%, 2S%); at the 25.9 pCi/l level (averaged over the 11 participants) is ±7.8 pCi/l (30.1%, 2S%); and at the 78.3 pCi/l level (averaged over the 11 participants) is ±20.8 pCi/l (26.6%, 2S%), for an average 2S percent of 30.0 percent for the range of iodine-131 concentrations of 7.8 to 78.3 pCi/l.

In terms of repeatability interval, on the basis of test error alone, the difference, in absolute value, of two test results obtained in the same laboratory at the 7.8 pCi/l level of iodine-131 concentration will be expected to exceed 1.9 pCi/l only 5 percent of the time; at the 25.9 pCi/l level, 6.3 pCi/l only 5 percent of the time; and at the 78.3 pCi/l level, 16.7 pCi/l only 5 percent of the time. This averages to be 23.4 percent of the iodine-131 concentration over the concentration range of 7.8 to 78.3 pCi/l.

In terms of reproducibility interval, on the basis of test error alone, the difference, in absolute value, of two test results obtained in different laboratories at the 7.8 pCi/l level of iodine-131 concentration will be expected to exceed 3.4 pCi/l only 5 percent of the time; at the 25.9 pCi/l level, 11.4 pCi/l only 5 percent of the time; and at the 78.3 pCi/l level, 30.2 pCi/l only 5 percent of the time. This averages to be 42.0 percent of the iodine-131 concentration over the concentration range of 7.8 to 78.3 pCi/l.

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The accuracy of the test procedure expressed as the accuracy index in percent for the three levels of iodine-131 concentrations (7.8, 25.9, and 78.3 pCi/l) are 97.4 percent, 97.3 percent, and 84.8 percent respectively, for an average accuracy index of 93.2 percent over the range of iodine-131 concentrations of 7.8 to 78.3 pCi/l.

The differences observed in the iodine carrier recoveries by the 11 participants (48 to 100 percent) showed a dependence on analyst technique for the method. Also, the similar average iodine carrier recoveries for the three samples (76.4, 79.3, and 79.8% respectively) does not offer any possible explanation for the low bias observed for test results of the 78.3 pCi/l sample.

The sensitivity of the test procedure is not limited as much by the chemistry of the procedure (iodine recovery), as it is by the background and the counting efficiency of the counting system used. With a beta background of less than 5 counts per minute (c/m) and a counting efficiency of more than 20 percent, the sensitivity of measurement required in the NIPDWR (1 pCi/l) can easily be met with a liter size sample. All participants, whose test results were used in the precision and accuracy analysis of this study, used counting instruments with beta backgrounds of less than 5 counts per minute and counting efficiencies of more than 20 percent.

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SECTION 3

RECOMMENDATIONS

It is recommended that the method tested in this study (Appendix B) be investigated for procedure steps that result in analyst technique dependence (as demonstrated by the variations in the iodine carrier recoveries). One possible weakness in the method is in the instructions for the reaction of solid zinc powder with solid silver iodide in an aqueous system.

The method should be modified to include a more detailed procedure for the preparation of palladium iodide-131 counting standard and for the determination of the counting efficiency. Also, a revision of section 6.5, the standardization of potassium iodate, is recommended.

The method should be re-tested with samples containing iodine-131 concentrations of 80 pCi/l and above to determine if the low bias is real. A thorough single-laboratory test of the method for that purpose should be sufficient to make that determination.

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SECTION 4

EXPERIMENTAL PROCEDURES

ANALYTICAL TEST PROCEDURE

The analytical test procedure (Method 902.0) that was tested in this study is given in detail in Appendix B.

COLLABORATIVE TEST PROCECEDURES

Iodine-131 Standard Solution Preparation

A standard iodine-131 solution was procured from Amersham Corporation. The certificate supplied with the solution indicated an activity of 61.4 μCi/4.947 g at 7:00 a.m. EST on November 21, 1979. The overall uncertainty was estimated to be less than +0.4 percent and -0.6 percent. The solution contained 0.1 mg/ml iodide carrier, 0.2 mg/ml sodium thiosulfate, and 1 mg/ml formaldehyde. A 1.0996 g portion of this standard was diluted to 100 ml with distilled and deionized water containing 0.2 g/l sodium thiosulfate pentahydrate and 0.035 g/l sodium carbonate. The total solution weighed 99.7857 g, and the iodide-131 concentration was 40.90 nCi/g (12/5/79, 7:00 a.m. EST). This solution was used to prepare the iodine-131 drinking water samples used in this collaborative study.

A 49.8086 g portion of the above solution was diluted to 100 ml with distilled and deionized water containing 0.2 g/l sodium thiosulfate pentahydrate and 0.035 g/l sodium carbonate. The total weight was 99.7041 g, and the iodine-131 concentration was 20.43 nCi/g (12/5/79, 7:00 a.m. EST). Sealed glass ampoules, containing 4 ml of this solution, were sent to participants of the collaborative study for use in checking the procedure and counting efficiency measurements.

Iodine-131 Drinking Water Sample Preparation

Drinking water samples containing 3 concentrations of iodine-131 were prepared in 82.5 l batches. The iodine-131 concentrations corrected for decay (to 1/14/80, 7:00 a.m. EST) were 7.8, 25.9, and 78.3 pCi/l. Unfiltered tap water which had been allowed to flow for a least 20 minutes was used to prepare the samples. The chloride and solids content of the tap water used for each batch were:

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	I-131 pCi/l	Cl ppm	Total Solids ppm	Suspended Solids ppm
Sample 1	7.8	50	510	0.022
Sample 2	25.9	51	506	0.022
Sample 3	78.3	49	483	0.022

Tap water, in about 2 kg amounts weighed to 0.1 g, was added into a 102 l polypropylene drum. Water quality control samples were taken before the first addition and at the mid- and end-points of the additions. After about 50 kg had been added, solutions containing 16.5 g of sodium thiosulfate pentahydrate (sufficient to give a concentration of 0.2 g/l for 82.5 l) and 2.8875 g of sodium carbonate (sufficient to give a concentration of 0.035 g/l for 82.5 l) were added. When the amount of tap water reached 65 kg, weighed solutions of iodine-131 (40.903 nCi/g) and lithium nitrate were added. The latter was sufficient to give a final lithium concentration of 3.33 ppm. The addition of tap water was continued to bring the total weight to 82.5 kg.

The solution was thoroughly mixed by use of a peristaltic pump whose intake was from the bottom of the tank. The solution was returned to the tank through a Viton® tube at about 2" below the liquid surface. Pumping was continued at a rate of 2.6 l/minute for 4 hours. After standing overnight, the solution was mixed again for an additional 5 hours of pumping. Lithium analyses by atomic absorption during and at the end of the second pumping period indicated complete mixing. About 3.5 kg portions of the iodine-131 drinking water batches were put into plastic cubetainers.

The density of the iodine-131 drinking water samples was 0.9992 g/cm^3 at 20.3°C. This value was the average of 2 measurements which were made in 2 different calibrated 100 ml volumetric flasks.

During transfer of the iodine-131 drinking water batches to sample containers, quality control fractions were taken at the initial, middle, and final stages of the transfer. These were analyzed for lithium to check for batch homogeneity. Also, the three quality control fractions taken from the sample 3 batch (78.3 pCi/l) were analyzed by Laboratory 1B for iodine-131 concentrations for another homogeneity check on that batch.

General Procedures

A total of 17 analysts from 16 laboratories agreed to participate in the collaborative study, and, of these 15 submitted their results. In addition, three more laboratories analyzed the iodine-131 drinking water samples by independent methods. All laboratories are listed in Appendix A. Each laboratory was supplied with the proposed EPA procedure (Appendix B) for the analysis of iodine-131 in drinking water, supplementary instructions and data sheet (Appendix C), a reference iodine-131 sample, and three 3.5 liter samples of drinking water containing 7.8, 25.9, and 78.3 pCi/l iodine-131 as of the collection date. (1/14/80, 7:00 a.m. EST).

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The reference and drinking water samples were prepared about 4 weeks before the collection date, (1/14/80), and participants were requested to analyze the samples on or soon after the collection date.

The results of each laboratory were reported on data sheets which specified sample size, gross counts, counting time, background rate, net c/m and d/m at midpoint of counting, counting efficiency, chemical recovery, and iodine-131 concentration at the collection date. All results involving calculations were checked, and any significant differences were resolved with the respective participants.

All participants in the collaborative study were encouraged to comment on all phases of the study. Several did so, and a summary of pertinent comments is included in Appendix D.

DATA PROCESSING PROCEDURES

The data from three participants were rejected for the following different reasons: (1) the samples were analyzed 24 days before the collection date. In this case the iodine-131 concentration at the time of analysis was over eight-fold greater than that for other participants, (2) due to a high background, all results were below the participants minimum sensitivity level of 290 pCi/l, and (3) the counting efficiency was questionable. A later determination indicated that this participant's initial counting efficiency was low by a factor of 2.1.

The data from the remaining 12 participants were tested for outliers by the ASTM-recommended criterion for rejection (ASTM, E-178, 1980). The results from one participant failed the test for each of the replicates for all three samples, and these results were rejected as outliers. For this rejection criterion, with n observations listed in order of increasing magnitude by $x_1 \leq x_2 \leq x_3 \leq \ldots \leq x_n$, if the outlying values x_n or x_1 are in question, then T_n or T_1 were calculated as follows:

$$T_n = (x_n - \bar{x})/s \qquad (1)$$

where: $T_1 = (\bar{x} - x_1)/s$

$T_n; T_1$ = test criterion

$\bar{x}$ = arithmetic average of all n values

s = the estimate of the population standard deviation based on the sample data

If T_n or T_1 values exceeded the critical value, the measurement in question was rejected. Critical values of T for a 1 percent two-sided level of significance were used for the rejection criterion.

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A statistical evaluation of the test results was done by the procedures described in E-691, E-177, and E-178 of the ASTM Standard Part 41, 1980. The standard deviations and other statistical parameters and equations for their calculations are listed below. The standard deviation of individual participant (or laboratory) test results, S_{ij}, was determined by Equation 2.

$$S_{ij} = \left[\sum_{h=1}^{n_{ij}} (X_{ijh}-\bar{X}_{ij})^2/(n_{ij}-1)\right]^{1/2} \quad (2)$$

where: X_{ijh} = the result reported for the h replicate of the j sample material by Lab i

$\bar{X}_{ij}$ = the mean of the individual results for sample j for Lab i

n_{ij} = the number of replicates reported for sample j by Lab i

The repeatability (within-laboratory) standard deviation, S_{r_j}, for each sample was determined by Equation 3.

Since the number of replicates is the same (3) for all participants for all three samples, the equation can be given as follows:

$$S_{r_j} = \left(1/P \sum_{i=1}^{P} S_{ij}^2\right)^{1/2} \quad (3)$$

where: P = the number of participants in the study.

The standard deviation of the grand average for each sample, $S_{\bar{X}_j}$, was determined by Equation 4.

$$S_{\bar{X}_j} = \left[\sum_{i=1}^{P} (\bar{X}_{ij} - \bar{X}_j)^2/(P-1)\right]^{1/2} \quad (4)$$

where: $\bar{X}_{ij}$ = the average of the test results for sample material j by Lab i

$\bar{X}_j$ = the grand average for sample material j

The standard deviation of between-laboratories precision for each sample material, S_{L_j}, was determined by Equation 5.

$$S_{L_j} = \left(S_{\bar{x}j}^2 - S_{r_j}^2/n\right)^{1/2} \quad (5)$$

The reproducibility (combined within- and between-laboratory) standard deviation for each sample, S_{R_j}, was determined by Equation 6.

$$S_{R_j} = (S_{r_j}^2 + S_{L_j}^2)^{1/2} \quad (6)$$

The deviation of laboratory averages from the grand average for each sample, d_{ij}, was determined by Equation 7.

$$d_{ij} = \bar{X}_{ij} - \bar{X}_j \quad (7)$$

The percent deviation of laboratory averages from the grand average for each sample, % d_{ij}, was determined by Equation 8.

$$\% \, d_{ij} = 100 \, (d_{ij}/\bar{X}_j) \quad (8)$$

The estimated standard error of a cell average, calculated on the basis of replication error only, U_j, for each sample is determined by Equation 9.

$$U_j = S_{r_j} [(P-1)/nP]^{1/2} \quad (9)$$

Reduced deviations (e_{ij}) for each participant for each sample were determined by Equation 10.

$$e_{ij} = d_{ij}/U_j \quad (10)$$

The ratio (k_{ij}) of the standard deviation of participant replicate analyses (S_{ij}) to the pooled standard deviation (S_{r_j}) for each sample was determined by Equation 11.

$$k_{ij} = S_{ij}/S_{r_j} \quad (11)$$

The coefficient of variation for repeatability (within-laboratory) for each sample, $V_{r_j}\%$, was determined by Equation 12.

$$V_{r_j}\% = 100 \, S_{r_j}/\bar{X}_j \quad (12)$$

The coefficient of variation for between-laboratories precision for each sample, $V_{L_j}\%$, was determined by Equation 13.

$$V_{L_j}\% = 100\, S_{L_j}/\bar{X}_j \quad (13)$$

The coefficient of variation for reproducibility (combined within- and between laboratory) for each sample, $V_{R_j}\%$, was determined by Equation 14.

$$V_{R_j}\% = 100\, S_{R_j}/\bar{X}_j \quad (14)$$

Smoothed values of the repeatability and reproducibility standard deviations for each sample, $\bar{S}_{r_j}$ and $\bar{S}_{R_j}$ were determined by Equations 15 and 16 respectively, using the respective average coefficients of variation.

$$\bar{S}_{r_j} = (\bar{V}_r\%)(\bar{X}_j)/100 \quad (15)$$

$$\bar{S}_{R_j} = (\bar{V}_R\%)\bar{X}_j/100 \quad (16)$$

Assuming that the test errors are normally distributed and using a 95 percent probability level, the 95 percent repeatability interval (I_{r_j}) and the 95 percent reproducibility interval (I_{R_j}) for each sample were determined using the smooth standard deviations in Equations 17 and 18 respectively.

$$I_{r_j} = 2(2)^{1/2}\, \bar{S}_{r_j} = 2.83\, \bar{S}_{r_j} \quad (17)$$

$$I_{R_j} = 2(2)^{1/2}\, \bar{S}_{R_j} = 2.83\, \bar{S}_{R_j} \quad (18)$$

The accuracy index, $A_j\%$, a percent relationship of the grand average, $\bar{X}_j$, to the known value, Y_j, for each sample material was determined by Equation 19.

$$A_j\% = 100\, \bar{X}_j/Y_j \quad (19)$$

A comparison of the grand average value, $\bar{X}_j$, with the known value, Y_j, for each sample in a bias test was determined by Equation 20 (Youden and Steiner, 1975).

$$t_j = \frac{\bar{X}_j - Y_j}{S_{\bar{X}_j}/(P)^{1/2}} \text{ , (P-1) degrees of freedom} \qquad (20)$$

where t_j = the bias test result for the j sample

P = the number of participants

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SECTION 5

RESULTS AND DISCUSSION

The three quality control fractions taken from the 78.3 pCi/l batch (sample 3) were analyzed for iodine-131 concentrations. The results are given in Table 8 and they show that the sample batch was homogeneous.

The quality control fractions (3 for each sample) for all three samples were checked for homogeneity by lithium analysis (by atomic absorption). Those results verified complete mixing of the three separate sample batches.

The test results from the 11 participants (10 laboratories) for the three iodine-131 sample concentrations are tabulated in Table 1. The participants are identified by randomly assigned numbers. Table 1 also lists the counting error (S_u), the iodine carrier recovery for each test result, and the grand average value for each sample.

Table 2 gives a complete summary of all precision parameters. The known value (Y_j) and grand average ($\bar{X}_j$), and the following precision parameters are listed for each sample:.

S_{x_j}, the standard deviation of the grand average for the j sample

S_{r_j}, the pooled standard deviation (within-laboratory) for the j sample

S_{L_j}, the square root of the component of variance between-laboratories, S_{L_j}, for the j sample

S_{R_j}, an estimate of the between-laboratories precision, a combined within- and between-laboratory precision, or reproducibility precision for the j sample

$V_{r_j}\%$, the coefficient of variation for repeatability, or the estimated relative standard deviation for within-laboratory precision, or repeatability index for the j sample

$V_{L_j}\%$, the coefficient of variation for between-laboratories precision for the j sample

$V_{R_j}\%$, the coefficient of variation for reproducibility, the percent coefficient of variation for the total variability of a single test result including both within- and between-laboratory variability, or reproducibility index for the j sample

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$\bar{S}_{r_j}$, the smoothed value of S_{r_j}

$\bar{S}_{R_j}$, the smoothed value of S_{R_j}

I_{r_j}, the 95 percent repeatability interval, the maximum permissible difference due to test error between two test results obtained in a single laboratory (or by a single participant) at the j sample level

I_{R_j}, the 95 percent reproducibility interval, the maximum permissible difference due to test error and systematic error between two test results obtained in two different laboratories at the j sample level

Table 3 lists by participant the average and the standard deviation of the participant test results for each of the three sample materials (cells). Table 3 also lists the S_{r_j} and U_j for each sample material.

Table 4 lists by participant the deviation and the percent deviation of the participant average from the grand average for each sample material and the standard deviation of the grand average for each sample material, $S_{\bar{x}_j}$.

Table 5 lists by participant the reduced deviations, e_j, for each sample material.

Table 6 lists by participant the ratio, k_j, of the participant test results standard deviation, S_{ij}, to the pooled standard deviation, S_{r_j}, for each sample material. This table points out potential outliers. Sample 2 for Participant 3 and Sample 1 for Participant 5 show ratio values higher than the critical value for 11 participants and 3 replicates (2.01). Since only one sample k_{ij} value for each of the two participants was higher than the critical value and the average of the k_{ij} values for all three sample materials for each of the two participants was lower than the critical value, all test results for the two participants were retained. Also, when the three replicate test results of the participant's sample of Table 6 that are higher than the critical value were tested by the ASTM T-test for individual replicate outliers, they all had lower values than the critical for 3 replicates at the 1 percent significance level. Therefore, they were not rejected.

Table 7 lists for each of the three sample materials the known value, (Y_j, pCi/l); the grand average, ($\bar{X}_j$, pCi/l); the accuracy index, (A_j, %); the standard deviation of the grand average, ($S_{\bar{x}_j}$, pCi/l); the bias value, (t_j); the critical value for significance for 11 participants, (t_c); and the number of participants in the study, (P_j). The table also shows the average accuracy index for the test procedure (93.2%). The bias test for Sample 3, the highest iodine-131 concentration of the three samples, gave a value (-4.14) which was higher in absolute value than the critical value (2.23). Therefore, the grand average for Sample 3 is significantly different from the known value for Sample 3 and therefore shows a low bias. The study does not show a significant bias for Samples 1 and 2, the two lower iodine-131 concentration levels (7.8 and 25.9 pCi/l respectively).

No explanation was evident or even suggested in the study for the low bias of the Sample 3 test results. The same amount of iodine carrier was used to determine recovery for the iodine-131 activity for all three samples and the recoveries are much the same for the three samples (averages of 76.4, 79.3, and 79.8% respectively). Iodine-131 has a very high specific activity, making its contribution to the iodine content of the sample very small compared to the iodine carrier added (8×10^{-10} µg/100 pCi compared to 10 mg/8 to 80 pCi).

From Table 2 it can be seen that the standard deviations (S_{r_j}, S_{L_j}, and S_{R_j}) depend on the level of iodine-131 concentration. However, the percent coefficients of variation as calculated from those standard deviations (V_{r_j} %, V_{L_j} %, and V_{R_j} %) show a rather fixed percent relationship to all three levels of iodine-131 concentration.

The repeatability (within-laboratory) and reproducibility (total between-laboratory) intervals (I_{r_j} and I_{R_j}) for each sample material were calculated with smoothed S_{r_j} and S_{R_j} values ($\bar{S}_{r_j}$ and $\bar{S}_{R_j}$ respectively). The values listed in Table 2 were calculated at the 95 percent probability level and are thereby designated as the 95 percent repeatability and 95 percent reproducibility intervals. The 95 percent repeatability interval is that difference value based on test error alone between two test results obtained by the same participant (or laboratory) that would be expected to be exceeded only about 5 percent of the time. The 95 percent reproducibility interval is that difference value between two test results obtained by two different participants (or laboratories) that would be expected to be exceeded only about 5 percent of the time. When difference values exceed the interval values, one or both test results are suspect. The study shows the estimated average 95 percent repeatability interval to be 23. percent of the iodine-131 concentration level over the range of 7.8 to 78.3 pCi/l . The estimated average 95 percent reproducibility interval is shown to be 42. percent over the same iodine-131 concentration range. It should be realized that those interval values are based on a limited 11 participant average.

The data that was rejected from four laboratories (for reasons footnoted in the tables) are tabulated in Appendix E in Tables E-1 through E-3.

The data that was submitted by three laboratories who used independent methods are tabulated in Appendix F in Tables F-1 through F-3.

Since the iodine-131 analysis is directly dependent on chemical recovery and counting efficiency, errors in these measurements will affect the results. In the procedure, 14.2 mg palladium iodide represents a 100 percent chemical recovery of iodine. A weighing error of 1.4 mg will result in a 10 percent error in iodine-131 analysis. Errors of this magnitude can easily occur due to extraneous matter and insufficient washing and drying. The same situation is encountered in the standardization of potassium iodate where the analysis also involves weighing about 14 mg of palladium iodide. A number of standardizations were carried out at Mound, and values ranging from 102 to 143 percent of the theoretical values were observed. Later determinations, corrected for blanks gave values close to theoretical values.

The determination of the counting efficiency for palladium iodide-131 was not completely defined in the procedure, and based on the experience of this work, this should be included in the procedure. Laboratories 14 and 17 found low counting efficiencies which resulted in high iodine-131 concentrations being reported. These laboratories determined the counting efficiency by adding iodine-131 to potassium iodate and carrying out the potassium iodate standardization according to Section 6.5 of procedure 902.0 (Appendix B). In these cases, the iodide-131 was probably oxidized by the potassium iodate and nitric acid to free iodine-131 which was partially lost during the heating interval preceding the addition of sulfite. At a later date, laboratory 17 redetermined their counting efficiency and found that their initial value was low by a factor of 2.1. This second measurement was carried out by the addition of iodide-131 to a known iodide solution and counting the precipitated palladium iodide-131.

TABLE 1. IODINE-131 IN WATER, REPLICATE ANALYSIS DATA

Lab	Sample, pCi/l($\pm S_u$)[a]R[b] 1	2	3
1A	7.7 (0.55) 82.3	27.3 (0.69) 85.0	60.8 (0.95) 88.4
	8.7 (0.43) 86.5	25.6 (0.65) 82.3	60.2 (1.00) 85.1
	7.7 (0.37) 87.9	26.7 (0.70) 80.8	62.0 (0.97) 85.1
1B	7.9 (0.46) 73.8	25.7 (0.65) 81.6	70.3 (1.10) 75.2
	8.0 (0.42) 72.3	26.6 (0.80) 77.3	68.8 (1.09) 81.6
	8.7 (0.47) 72.3	27.1 (0.73) 76.6	74.7 (1.16) 70.9
2	8.2 (0.13) 85.6	25.2 (0.21) 91.4	75.7 (0.38) 87.8
	7.3 (0.13) 89.9	25.4 (0.22) 85.6	75.6 (0.38) 87.8
	8.1 (0.14) 84.9	26.0 (0.22) 89.9	74.7 (0.39) 84.2
3	8.8 (0.55) 88.1	22.5 (0.83) 83.9	67.7 (1.65) 59.5
	8.8 (0.62) 76.2	30.2 (1.32) 50.6	84.3 (2.14) 48.2
	10.2 (0.86) 51.8	35.8 (1.40) 52.4	67.9 (1.71) 64.3
4	5.6 (0.88) 82.2	20.6 (1.16) 91.5	67.9 (1.74) 97.7
	6.4 (0.85) 89.2	25.0 (1.30) 86.9	67.9 (2.04) 93.5
	6.3 (0.83) 77.7	22.6 (1.06) 94.0	76.5 (2.20) 86.2
5	6.9 (0.51) 89.9	28.0 (0.90) 81.6	73.9 (1.32) 89.9
	8.3 (0.60) 80.2	23.2 (0.81) 89.4	63.2 (1.28) 86.8
	5.0 (0.49) 90.8	20.9 (0.95) 66.3	53.6 (1.17) 92.2

(continued)

TABLE 1. (Continued)

Lab	Sample, pCi/l(±S_u)[a]R[b] 1			2			3		
7	8.0	(0.44)	62.7	24.2	(0.83)	66.2	48.5	(0.93)	92.3
	8.3	(0.46)	64.8	24.8	(0.87)	68.3	51.5	(3.00)	63.4
	7.8	(0.49)	64.1	24.1	(0.78)	68.3	61.3	(1.61)	43.7
9	4.9	(0.68)	73.1	19.0	(1.03)	69.2	57.6	(1.46)	86.9
	5.1	(0.69)	72.3	17.7	(0.84)	100.0	59.7	(1.53)	80.0
	5.4	(0.75)	65.4	19.4	(0.99)	75.4	59.9	(1.49)	90.8
11	7.8	(0.54)	63.0	27.0	(0.82)	77.5	60.5	(1.17)	87.0
	8.2	(0.55)	70.3	27.0	(0.84)	76.1	58.3	(1.17)	83.3
	7.9	(0.47)	79.0	29.0	(0.89)	70.3	59.6	(1.22)	77.5
15	8.3	(0.45)	85.6	30.9	(0.75)	85.9	85.1	(1.21)	84.5
	9.3	(0.47)	83.1	28.2	(0.71)	88.7	84.1	(1.21)	84.5
	8.8	(0.45)	90.1	29.3	(0.73)	87.3	87.1	(1.23)	84.5
16	7.7	(0.66)	62.1	25.1	(0.98)	71.2	54.0	(1.42)	69.1
	7.5	(0.76)	50.8	21.7	(0.84)	83.2	57.5	(1.43)	72.6
	6.8	(0.58)	72.6	19.7	(0.82)	81.8	60.6	(1.51)	68.4
average	7.6		76.4	25.2		79.3	66.4		79.8

[a] S_u - counting uncertainty, pCi/l, one sigma

[b] R - recovery, %

TABLE 2. IODINE-131 IN WATER, PRECISION SUMMARY

Parameter[a]	Samples 1	2	3	Average
Y_j (pCi/l)	7.8	25.9	78.3	
$\bar{X}_j$ (pCi/l)	7.6	25.2	66.4	
$S_{\bar{x}_j}$ (pCi/l)	1.19	3.25	9.54	
S_{r_j} (pCi/l)	0.66	2.64	5.14	
S_{L_j} (pCi/l)	1.14	2.87	9.07	
S_{R_j} (pCi/l)	1.32	3.90	10.43	
V_{r_j}%	8.7	10.5	7.7	9.0
V_{L_j}%	15.0	11.4	13.7	13.4
V_{R_j}%	17.4	15.5	15.7	16.2
$\bar{S}_{r_j}$ (pCi/l)	0.676	2.243	5.910	
$\bar{S}_{R_j}$ (pCi/l)	1.221	4.048	10.665	
I_{r_j} (pCi/l)	1.9	6.3	16.7	
I_{R_j} (pCi/l)	3.4	11.4	30.2	

[a]Terms defined in text and the List of Abbreviations and Symbols

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TABLE 3. IODINE-131 IN WATER, CELL AVERAGES ($\bar{X}_{ij}$) AND CELL STANDARD DEVIATIONS (S_{ij})

	Sample					
	1		2		3	
Lab	$\bar{X}_{ij}$	S_{ij}	$\bar{X}_{ij}$	S_{ij}	$\bar{X}_{ij}$	S_{ij}
1A	8.0	0.57	26.5	0.83	61.0	0.93
1B	8.2	0.42	26.5	0.73	71.3	3.06
2	7.9	0.49	25.6	0.42	75.3	0.56
3	9.3	0.80	29.5	6.71	73.3	9.53
4	6.1	0.41	22.7	2.20	70.8	4.94
5	6.7	1.66	24.0	3.65	63.6	10.17
7	8.0	0.28	24.3	0.39	53.8	6.72
9	5.1	0.26	18.7	0.91	59.0	1.28
11	8.0	0.21	27.7	1.20	59.5	1.11
15	8.8	0.47	29.5	1.33	85.4	1.49
16	7.3	0.48	22.2	2.76	57.4	3.31
$\bar{X}_j$, column average	7.6		25.2		66.4	
S_{r_j}	0.66		2.64		5.14	
U_j	0.363		1.453		2.830	

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TABLE 4. IODINE-131 IN WATER, DEVIATIONS FROM AVERAGES (d_{ij}, pCi/l) AND PERCENT DEVIATIONS FROM AVERAGE (% d_{ij})

Lab	Sample 1		Sample 2		Sample 3	
	d_{ij}	% d_{ij}	d_{ij}	% d_{ij}	d_{ij}	% d_{ij}
1A	0.40	5.26	1.33	5.28	5.4	-8.13
1B	0.61	8.05	1.26	5.00	4.9	7.38
2	0.30	3.95	0.36	1.43	8.9	13.40
3	1.67	22.03	4.29	17.03	6.9	10.39
4	-1.50	-19.79	-2.49	-9.88	4.4	6.63
5	-0.87	-11.48	-1.15	-4.57	-2.8	-4.22
7	0.44	5.80	-0.86	-3.41	-12.6	-18.98
9	-2.46	-32.45	-6.47	-25.68	-7.4	-11.14
11	0.39	5.15	2.47	9.81	-6.9	-10.39
15	1.20	15.83	4.27	16.95	19.0	28.61
16	-0.26	-3.43	-3.01	-11.95	-9.0	-13.55
$S_{\bar{x}_j}^2$	1.4239		10.5407		91.032	
$S_{\bar{x}_j}$	1.1933		3.2466		9.5411	

[a,b]Terms defined in text and List of Abbreviations and Symbols

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TABLE 5. IODINE-131 IN WATER, REDUCED DEVIATIONS, e_{ij}[a]

Lab	Sample 1	Sample 2	Sample 3
1A	1.10	0.92	-1.91
1B	1.68	0.88	1.73
2	0.83	0.25	3.15
3	4.60	2.95	2.44
4	-4.13	-1.71	-1.56
5	-2.39	-0.79	-0.99
7	1.21	-0.59	-4.45
9	-6.77	-4.45	-2.62
11	1.07	1.70	-2.44
15	3.30	2.94	6.72
16	-0.72	-2.07	-3.18
U_j[b]	0.363	1.453	2.830
S_{r_j}	0.66	2.64	5.14

a,bTerms defined in text and List of Abbreviations and Symbols

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TABLE 6. IODINE-131 IN WATER, RATIO OF STANDARD DEVIATIONS TO POOLED STANDARD DEVIATION, k_{ij}[a]

Lab	Sample 1	Sample 2	Sample 3
1A	0.86	0.31	0.18
1B	0.64	0.28	0.60
2	0.74	0.16	0.11
3	1.21	2.54	1.85
4	0.62	0.83	0.95
5	2.45	1.38	1.98
7	0.42	0.15	1.30
9	0.39	0.35	0.25
11	0.38	0.45	0.21
15	0.71	0.50	0.29
16	0.73	1.05	0.64

[a]Term defined in text and List of Abbreviations and Symbols

TABLE 7. IODINE-131 IN WATER, ACCURACY INDEX (A_j%) AND BIAS (t_j)

Parameter[a]	Sample 1	Sample 2	Sample 3	Average
Y_j, pCi/l	7.8	25.9	78.3	
$\bar{X}_j$, pCi/l	7.6	25.2	66.4	
A_j, %	97.4	97.3	84.8	93.2
$S_{\bar{x}_j}$, pCi/l	1.19	3.25	9.54	
t_j	-0.55	-0.71	-4.14	
t_c	2.23	2.23	2.23	
P	11	11	11	

[a]Terms defined in text and List of Abbreviations and Symbols.

TABLE 8. IODINE-131 IN WATER, SAMPLE 3 QUALITY CONTROL DATA

Sample	I-131 pCi/l ($\pm S_\mu$)[a]	$\bar{X}_{ij}$[b] (pCi/l)	S_{ij}[c] (pCi/l)	Accuracy[d] Index, A%	Chemical Recovery (%)
QC-1	69.1 (1.17)				89.4
	72.4 (1.16)	69.1	3.26	88	90.1
	65.9 (1.08)				92.9
QC-2	69.1 (1.30)				68.1
	69.8 (1.17)	68.0	2.47	87	79.4
	65.2 (1.13)				78.7
QC-3	70.7 (1.24)				69.5
	68.8 (1.09)	69.6	1.05	89	82.3
	69.1 (1.16)				72.3

[a,b,c,d]Terms defined in text and List of Abbreviations and Symbols.

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TABLE 9. IODINE-131 IN WATER, TEST SAMPLE PREPARATION DATA

	Batch Dilutions			
Sample Batch	Aliquot Weight (g)	Concentration (nCi/g)	Final Volume (l)	Final Concentration (pCi/l)
1	0.4922	40.903	82.7	244 (7.76)[a]
2	1.6672	40.903	83.8	814 (25.9)[a]
3	4.9663	40.903	82.7	2462 (78.3)[a]

[a] Concentration values in parentheses have been decay-corrected from the 12/5/79 preparation date (date for the 40.903 nCi/g concentration) to the designated collection date of 1/14/80.

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REFERENCES

American Society for Testing and Materials, 1980 Annual Book of Standards, Part 41, Designation: E177-71 (Reapproved 1980) p. 195; E178-80, p. 206; and E691, p. 959.

Youden, W. J. and E. A. Steiner, "Statistical Manual of the Association of Official Analytical Chemists," Association of Official Analytical Chemists, Washington, DC, 1975.

EPA-600/4-75-008 (Revised) March 1976, "Interim Radiochemical Methodology for Drinking Water".

EPA-600/4-80-032 August 1980, "Prescribed Procedures for Measurement of Radioactivity in Drinking Water".

EPA-570/9-76-003, "National Interim Primary Drinking Water Regulations".

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APPENDIX A

LABORATORIES PARTICIPATING IN THE IODINE-131 DRINKING WATER COLLABORATIVE STUDY

Alabama State Department of Health
Montgomery, Alabama

California State Department of Health Services
Sanitation and Radiation Laboratory
Berkeley, California

Colorado State Department of Health
Denver, Colorado

New York State Department of Health
Albany, New York

North Carolina State Laboratory of Public Health
Raleigh, North Carolina

Kentucky Department of Human Resources
Frankfort, Kentucky

Louisiana Department of Health and Human Resources
New Orleans, Louisiana

Orlando Laboratories, Inc.
Orlando, Florida

Texas State Department of Health
Austin, Texas

U.S. Army
Walter Reed Army Medical Center
Washington, D.C.

U.S. Department of Energy
Environmental Measurements Laboratory
New York, New York

U.S. Environmental Protection Agency
Eastern Environmental Radiation Facility
Montgomery, Alabama

U.S. Environmental Protection Agency
Environmental Monitoring and Support Laboratory
Cincinnati, Ohio

U.S. Food and Drug Administration
Winchester Engineering and Analytical Center
Winchester, Massachusetts

University of Pittsburgh
Graduate School of Public Health
Pittsburgh, Pennsylvania

Washington State Office of Public Health
Laboratories and Epidemiology
Seattle, Washington

Yankee Atomic Electric Company
Westborough, Massachusetts

APPENDIX B

RADIOACTIVE IODINE IN DRINKING WATER*
METHOD 902.0

1. Scope and Application

   1.1 According to the National Interim Primary Drinking Water Regulations (NIPDWR), drinking water supplies designated as possibly contaminated by effluents from nuclear facilities are to be monitored quarterly for iodine-131. Also, for drinking water supplies not designated as above, when the gross beta activity exceeds 50 pCi/l, the drinking water must be analyzed for specific radionuclides for the assignment of dose for people using those supplies. This method covers the measurement of radioactive iodine in a sample of drinking water.

   1.2 The NIPDWR lists a required sensitivity of measurement of 1 pCi/l for iodine-131 in drinking water. This method provides for the separation and concentration of iodine from sufficiently large samples so the 1 pCi/l required sensitivity can be met. See Appendix C for calculations of necessary counting time to meet the required sensitivity of measurement.

2. Summary of Method

   2.1 Stable iodate carrier is added to an acidified sample of drinking water. The iodate is reduced to the iodide state with Na_2SO_3 and all iodine activity is precipitated as AgI. The precipitate is dissolved and purified with zinc powder and sulfuric acid and reprecipitated as PdI_2 for counting.

   2.2 The final precipitate is counted either with a beta system or beta/gamma coincidence scintillation system. Since there is very little likelihood of any other iodine isotope but iodine-131 present in drinking water, gamma-ray spectrometry is not necessary, but can be employed to ascertain the purity of the separation.

*This procedure was prepared by the U.S. EPA, Cincinnati, Ohio. Appendices cited are not included in this report.

3. Sample Handling and Preservation

3.1 (See Section 3.1, Method 900.0).

3.2 The drinking water sample should not have acid added at time of collection, and radiochemical analysis for iodine-131 should be performed soon after receipt to minimize radioactive decay losses ($t_{\frac{1}{2}}$ = 8.04 d).

4. Interferences

4.1 The presence of stable iodide in the drinking water would interfere by indicating a higher chemical yield than actually resulted.

4.2 Counting efficiencies should be determined for the range of precipitate weights anticipated particularly if beta/gamma counting is performed.

5. Apparatus - See Appendix D for details and specifications.

5.1 Gamma spectrometer system, or

5.2 Gas-flow proportional counting system. (Low background Beta <3 c/m), or

5.3 Beta/Gamma coincidence scintillation system.

5.4 Plastic ring and disc mounts

5.5 Mylar film

5.6 Teflon filter holder

5.7 Electric hotplate

5.8 Analytical balance

5.9 pH meter

5.10 Drying oven

5.11 Aluminum desiccator

5.12 Centrifuge

5.13 Glassware

6. Reagents

6.1 Deionized distilled water.

6.2 Ammonium hydroxide, 6N: Mix 2 volumes 15N NH OH (conc.) with 3 volumes water.

6.3 Ethanol, 95 percent: C_2H_5OH

6.4 Hydrochloric acid, 6N: Mix 1 volume 12N HCl (conc.) with 1 volume water.

6.5 Iodate carrier, (IO_3^-), 10 mg/ml (as I^-), standardized: Dissolve 1.685 g KIO_3 in water and dilute to 100 ml. Store in a dark flask.

Standardization: (in triplicate)

Pipette 1.0 ml potassium iodate (KIO_3) carrier into a 50 ml centrifuge tube containing 10 ml 0.4N HNO_3. Heat and stir in a water bath, add 1 ml 1M Na_2SO_3 and digest for 5 minutes. Add 1 ml 0.2M $PdCl_2$ and stir to precipitate and coagulate PdI_2. Cool and filter through a tared fritted glass funnel (fine to medium). Dry, cool, and weigh as PdI_2.

6.6 Nitric acid, 16N: HNO_3 (conc.) sp. gr. 1.42, 70.4 percent.

6.7 Nitric acid, 0.4N: Mix 1 volume 16N HNO_3 (conc.) with 39 volumes of water.

6.8 Palladium chloride, $PdCl_2$, 5 percent solution in 0.6N HCl: reagent grade.

6.9 Palladium chloride, 0.2M: a) Dissolve 3.542 g $PdCl_2$ in 100 ml 1N HCl or b) Mix 118 ml 5 percent $PdCl_2$* with 50 ml 2N NCl.

6.10 Silver nitrate, 0.1M: Dissolve 1.7 g $AgNO_3$ in water and dilute to 100 ml. Store in a dark flask.

6.11 Sodium sulfite, 1M: Dissolve 5 g Na_2SO_3 in 40 ml water. Prepare fresh reagent every week.

6.12 Sulfuric acid, 2N: Mix 1 volume 36N H_2SO_4 (conc.) with 17 volumes of water.

6.13 Zinc powder: reagent grade.

*Palladium chloride, 5 percent in hydrochloric acid is available as a reagent solution from several chemical houses, and the 4 oz. size (118 ml) can be diluted directly to 0.2M by adding 50 ml 2N HCl to the stock reagent.

7. Calibrations

7.1 The detection efficiency of the gamma ray spectrometer must be obtained for measuring the iodine-131 photopeak (364 kev). With commercially available calibrated standards, geometric configurations are to be prepared comparable to the final separated sample so the cpm per dpm factors can be determined.

7.2 Similar calibrations are to be made for the beta and the beta/gamma coincidence counters.

8. Procedure

8.1 To a 1000-ml drinking water sample, add 7.5 ml 16N HNO_3 and 1.0 ml iodate (IO_3^-) carrier. Mix well.

8.2 Add 4 ml freshly prepared 1M Na_2SO_3 and stir for 30 minutes.

8.3 Add 20 ml 0.1M $AgNO_3$, stir for 1 hour, and settle for another hour.

8.4 Decant and discard as much as possible of the supernatant. Filter the remainder through a glass-fiber filter and discard filtrate.

8.5 Transfer the filter to a centrifuge tube and slurry with 10 ml water. Add 1 gram zinc powder and 2 ml 2N H_2SO_4, and stir frequently for at least 30 minutes.

8.6 Filter, with vacuum, through a fine-fritted glass funnel and collect filtrate in an Erlenmeyer flask. Use a few ml water to wash both residue and filter and add the wash to filtrate in the flask. Discard residue.

8.7 Add 2 ml 6N HCl to the filtrate and heat. Add 1 ml 0.2M $PdCl_2$ and digest for at least 5 minutes. Centrifuge and discard supernatant.

8.8 Dissolve the precipitate in 5 ml 6N NH_4OH and heat in boiling water bath. Filter through a glass-fiber filter and collect filtrate in a centrifuge tube. Discard filter and residue.

8.9 Neutralize the filtrate with 6N HCl, add 2 ml additional, and heat in a water bath. Add 1 ml 0.2M $PdCl_2$ to reprecipitate PdI_2 and digest for 10 minutes. Cool slightly.

8.10 Transfer to a tared glass-fiber filter with water. Wash successively with 5 ml portions of water and ethanol.

8.11 Dry to constant weight at 105°C, cool, mount, and count either gamma, beta or beta/gamma coincidence.

Note: Ordinarily, the final PdI_2 precipitate is collected on a glass-fiber filter and counted in a G-M system where the natural background is about 0.8 c/m. Glass-fiber filters add about 0.5 c/m to the background so that the PdI_2 counted this way will have about a 1.3 c/m background and a counting efficiency of 35 percent. However, if the precipitate is collected on an 0.8-μ membrane filter, and dried for 30 minutes at 70°C, it is possible to count the PdI_2 in a beta/gamma coincidence scintillation system where the background coincidence count is less than 0.1 c/m and the counting efficiency is greater than 40 percent (Brauer, et al.).

9. Calculations

9.1 Calculate the iodine concentration, D, in picocuries per liter as follows:

$$D = \frac{C}{2.22 \times EVR \times A}$$

where:

C = net count rate, cpm,
E = counter efficiency,
V = liters of sample used,
R = fractional chemical yield,
A = decay correction for iodine-131 ($t_{\frac{1}{2}}$ = 8.04 d),
2.22 = conversion factor from disintegrations/minute to picocuries

9.2 For calculations involving gamma-ray spectrometry:

a) Calculate the counter efficiency, E, as follows:

$$E = Fp$$

where:

F = fractional abundance of the gamma ray, gammas/disintegration,
P = photopeak detection efficiency, counts/gamma ray.

b) Calculate decay corrections as follows:

$$A = A_0 e^{-0.693t/T}$$

where:

A = relative activity at time t,
A_0 = initial activity
e = base of natural logarithms
t = elapsed time from collection to counting, and
T = half-life of iodine-131 (8.04 d), in same units as t

10. Precision and Accuracy

10.1 Following replicate analyses performed by three operators on aliquots containing 5000 dpm/l, the accuracy and precision of this procedure were determined.

The accuracy of triplicate analyses on 1000 ml aliquots was ±4 percent of the expected value. The precision attained by the technicians based on three sets of triplicate analyses was ±2 percent.

Bibliography

1. Brauer, F. P., J. H. Kaye, and R. E. Connaly, X-ray and B-γ Coincidence Spectrometry Applied to Radiochemical Analysis of Environmental Samples. Advances in Chemistry Series, No. 93, Radionuclides in the Environment, pp. 231-253. American Chemical Society (1970).

2. Kleinberg, J., and G. A. Cowan. The Radiochemistry of Fluorine, Chlorine, Bromine, and Iodine. AEC Rept. NAS-NS-3005 (1960).

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APPENDIX C

INSTRUCTIONS AND DATA SHEET

MONSANTO RESEARCH CORPORATION

From (Name and Location): Mound Facility - Miamisburg, Ohio

cc: Earl Whitaker, EPA Las Vegas

Date : November 30, 1979

Subject : Iodine-131 Collaborative Study

Reference: Sept. 21, 1979 Letter of C. T. Bishop

TO : All Participants

The samples for the iodine-131 in drinking water will be sent to you near the end of December, 1979. As indicated earlier we will be sending three water samples each having different concentrations of iodine-131, and we are requesting triplicate analysis of each sample. We are requesting that the samples be analyzed after January 1, 1980 and preferably the week of January 14. Because of the 8-day half-life of iodine-131, it will be necessary that all samples be analyzed by January 25 at the latest. Iodine-131 concentrations (as of 1/1/80) will be less than 300 pCi/l in each of the three samples.

An iodine-131 standard containing ~0.1 μCi and a revised procedure will be sent to you shortly. The standard may be used to calibrate your instrumentation and/or to prepare your own trial samples to test the procedure. If there are any special reagents that you need, please let us know now so that they can be sent to you. Since a few significant changes have been made in the procedure sent to you earlier, it is suggested that you destroy the procedure sent to you in September and follow the instructions exactly as they are given in the revised procedure dated 11/19/79.

To ensure compliance with NRC regulations, we must have written verification that you are licensed to receive the 0.1 μCi of the I-131 standard. Please complete the enclosed license certification form and return it to us as soon as possible. If you have questions concerning this form or other matters of this study, please call us.

Carl T. Bishop
FTS 774-3927 or (513) 865-3927

William H. Yanko
FTS 774-3930 or (513) 865-3930

/km

BY-PRODUCT LICENSE CERTIFICATION

_____

(Institution)

_____

(Address)

is authorized to receive 0.1 microcuries of Iodine-131 under license

_____

(State or NRC) (No.) (Expiration Date)

_____

(Radiation Safety Officer)

_____

(Date)

Send to:

William H. Yanko
Monsanto Research Corporation
Mound Facility
Miamisburg, OH 45342

MONSANTO RESEARCH CORPORATION

From (Name and Location): Mound Facility - Miamisburg, OH 45342 *cc:* Earl Whittaker
EPA, Las Vegas

Date : December 18, 1979

Subject : Iodine-131 Collaborative Study

Reference: Nov. 30, 1979 Memo of C. T. Bishop
and W. H. Yanko

TO : All Participants

Enclosed is a copy of the procedure "Radioactive Iodine in Drinking Water, Method 902.0", dated 11/19/79, to be used in the collaborative study. Also enclosed are the "Collaborative Study Instructions, I-131 in Drinking Water - January, 1980" and two data sheets.

Samples and the iodine-131 standard will have been shipped by December 20; if you have not received your samples by January 7, 1980 please let us know.

We would like to thank your laboratory for participating in this study, and we will send you preliminary results of the study within a month or two after all of the data are received. We are requesting that data be submitted by January 28, 1980. If you have any questions at this time or when you begin analyzing the samples, please do not hesitate to call.

Carl T. Bishop
FTS 774-3927 or (513) 865-3927

William H. Yanko
FTS 774-3930 or (513) 865-3930

/km

MRC-ML-6443 (6-78)
17-1170

COLLABORATIVE STUDY INSTRUCTIONS
I-131 IN DRINKING WATER - JANUARY, 1980

1. Three samples with different I-131 levels will be provided in 1-gallon cubetainers. Each will contain over 3.1 liters of solution which is sufficient for triplicate analyses of 1 liter samples. The maximum I-131 level will be less than 300 pCi/L as of January 1, 1980, 7:00 a.m. EST, the collection date specified on the data sheet. If possible, we prefer that the I-131 analyses be conducted the week of January 14, 1980.

2. Tap water, containing 0.2 g/L sodium thiosulfate, $Na_2S_2O_3 \cdot 5H_2O$, and 0.035 g/L Na_2CO_3, was used in preparing all samples. The density of each sample is 0.9992 g/cm^3 at 20.3 °C.

3. Use the revised (11/19/79) EPA procedure (method 902.0) which is enclosed. The following changes were made in the procedure sent to you in September:

   a. Step 6.5: Standardization of KIO_3

      Line 2 - use 10 ml 0.4N HNO_3 instead of 0.1N HNO_3.

      Line 3 - Use 1 ml 1M Na_2SO_3 instead of 2 ml.

      These changes are important since no PdI_2 will precipitate using the original directions.

   b. 6.7: Palladium Chloride, 0.2M

      The directions as specified will give a 0.2M solution of $PdCl_2$ in 1N HCl since the commercial 5% $PdCl_2$ solution contains 2% HCl.

      If you use crystalline $PdCl_2$, prepare your 0.2M solution with 1N HCl.

   c. 8.1: Use 1000 ml (instead of 2000 ml), add 7.5 ml 16 N HNO_3, and 1 ml iodate carrier.

   d. 9.1 and 9.2 (b): Use 8.04 days for the half-life of I-131.

4. An ampoule of I-131 of concentration known to be about ±1% (∿ 4 g, specific activity 20.43 nCi/g as of 12/5/79; 7:00 a.m. EST) is being sent to you. You may wish to use this standard for test runs or to calibrate your counting instrumentation.

5. If needed, Mound will supply any of the special reagents required for the analysis. Contacts are given below.

6. Two copies of the data sheet for your analyses are enclosed. Please complete one and return to Mound Facility by January 28, 1980. Please note your ID designation. Your results will be reported under this ID, and your laboratory will not be identified with your data.

7. Individual counting data is requested to resolve counting uncertainties from other errors. Gross counts and d/m specified on the data sheet are defined:

    a. Gross counts - total counts including background.

    b. d/m I-131 at midpoint of counting interval - c/m corrected for counting efficiency and chemical recovery.

8. Any comments you have on the procedure, positive or negative, would be appreciated when results are submitted.

9. A limited number of the three water samples or the I-131 standard are available, if one of your samples is spilled, contaminated, etc.

If you have any questions contact:

Bill Yanko	513-865-3930 (commercial) or 774-3930 (FTS)
Carl Bishop	513-865-3927 (commercial) or 774-3927 (FTS)

APPENDIX C
DATA SHEET

11/30/79

I-131 IN DRINKING WATER
Collaborative Tests (January, 1980)

Laboratory _____
Contact Person _____
Collection Date: _____

Sample	Size (ml)	Gross Counts[a]	Counting Time (min)	Bkgd. c/m	I-131 at Mid Point of Counting Time c/m	I-131 at Mid Point of Counting Time d/m[b]	I-131 at Mid Point of Counting Time Date	I-131 at Mid Point of Counting Time Time	Counting Efficiency %	Chemical Recovery %	I-131 at Collection Date pCi/L
1 a											
1 b											
1 c											
2 a											
2 b											
2 c											
3 a											
3 b											
3 c											

a. Total counts including background.
b. d/m (c/m corrected for counting efficiency and chemical recovery).

Please send results to W. H. Yanko, Monsanto Research Corp., Mound Facility, Miamisburg, OH 45342, by January 28, 1980.

APPENDIX D

COMMENTS FROM PARTICIPANTS IN THE COLLABORATIVE STUDY

1. Procedure 902.0 (Appendix 2) implied that the palladium iodide-131 counting standard for the determination of the counting efficiency can b prepared using the method for the standardization of potassium iodate according to Section 6.5. Use of this method gave erroneously low counting efficiency values. Correct values were obtained by use of iodide carrier instead of potassium iodate. The procedure should be modified to include a detailed method for the determination of the counting efficiency.

2. Drying of palladium iodide in a vacuum over phosphorus pentoxide was preferred to drying at 105°C.

3. The 40 percent counting efficiency for beta/gamma coincidence counting claimed in Section 8.11 was not attained.

4. The counting efficiency determinations were not reproducible and the range was relatively large. Variations could be due to the variant size of the palladium iodide or loss of iodide-131 caused by heat during drying of the material.

5. All heating periods in Section 6.5 should be specified.

6. Since problems were encountered with the procedure and the determination of the counting efficiency of the prepared palladium iodide-131 counting standard, the results were not submitted.

7. Activity of the iodine-131 standard supplied to all participants was measured 2 months after the preparation date and 19.4 ± 1.3 nCi/g was found. This was good agreement with the specified 20.4 nCi/g, considering the age of the sample.

8. Centrifugation instead of filtration was suggested for steps 8.4 and 8.6. A test of this modification gave 90 percent chemical yields.

9. In step 8.5, it is easier to use a small beaker for the reduction with zinc powder and to use Millipore filter paper for the filtration.

10. It is more convenient to use a tared glass-fiber filter in step 6.5 instead of a fritted glass funnel.

APPENDIX E

TABLE E-1. IODINE IN WATER, REJECTED DATA, SAMPLE 1 (7.8 pCi/l)

Lab	I-131 pCi/l ($\pm S_\mu$)[a]	$\bar{X}_{ij}$[b] (pCi/l)	S_{ij}[c] (pCi/l)	Accuracy[d] Index, A	Chemical Recovery (%)
8	no data				
6*	9.8 (0.29)				90.0
	10.1 (0.31)	10.3	0.63	133	85.0
	11.0 (0.31)				85.0
14†	14.0 (2.38)				85.0
	16.7 (2.75)	15.6	1.44	201	75.0
	16.2 (3.12)				64.0
17‡	16.8 (0.78)				62.7
	17.1 (0.49)	16.3	0.64	213	62.0
	15.8 (0.83)				60.6
20*	8.7 (0.13)	9.4	0.90	121	76.9
	10.0 (0.13)				79.7

[a] S_μ - Uncertainty due to counting (1 sigma)

[b] $\bar{X}_{ij}$ - Average of replicates

[c] S_{ij} - Experimental (within-laboratory) standard deviation

[d] A - 100 $\bar{X}_{ij}$/known, (Y_j)

A_j - 100 $\bar{X}_j/Y_j$

*Rejected because analysis was made before the reference date

†Rejected by ASTM outlier test

‡Rejected due to error in counting efficiency

TABLE E-2. IODINE-131 IN WATER, REJECTED DATA, SAMPLE 2 (25.9 pCi/l)

Lab	I-131 pCi/l (±Sμ)[a]	$\bar{X}_{ij}$[b] (pCi/l)	S_{ij}[c] (pCi/l)	Accuracy[d] Index, A	Chemical Recovery (%)
6*	40.8 (0.57)				91.0
	40.5 (0.57)	39.1	2.67	151	90.0
	36.0 (0.65)				63.0
8**	$\bar{<}$ 290				43.3
	$\bar{<}$ 290				41.4
	$\bar{<}$ 290				44.8
14†	47.3 (3.39)				83.0
	55.7 (4.19)	49.4	5.51	191	66.0
	45.3 (3.57)				76.0
17‡	60.0 (1.89)				44.4
	54.8 (2.15)	54.6	5.55	210	74.7
	48.9 (1.57)				75.4
20*	29.2 (0.46)			113	81.8

[a,b,c,d]See Table E-1 footnotes

*Rejected because analysis was made before reference date

**Rejected because of high background

†Rejected by ASTM outlier test

‡Rejected due to error in counting efficiency

TABLE E-3. IODINE-131 IN WATER, REJECTED DATA, SAMPLE 3 (78.3 pCi/l)

Lab	I-131 pCi/l (±Sμ)[a]	$\bar{X}_{ij}$[b] (pCi/l)	S_{ij}[c] (pCi/l)	Accuracy[d] Index, A	Chemical Recovery (%)
6*	91.8 (1.20)				95.0
	88.3 (1.47)	89.0	2.51	114	94.0
	87.0 (1.59)				80.0
8**	$\overline{<}$ 290				45.6
	$\overline{<}$ 290				61.0
	$\overline{<}$ 290				80.0
14†	168.9 (5.78)				81.0
	153.5 (5.51)	157.7	9.81	201	81.0
	150.8 (5.55)				82.0
17‡	145.7 (1.13)				73.3
	163.3 (1.90)	149.9	11.96	191	77.5
	140.5 (2.53)				74.7
20*	92.0 (0.21)			117	79.0

a,b,c,d See Table E-1 footnotes

*Rejected because analysis was made before reference date

**Rejected because of high background

†Rejected by ASTM outlier test

‡Rejected due to error in counting efficiency

APPENDIX F

TABLE F-1. RESULTS OF LABS USING INDEPENDENT METHODS FOR IODINE-131 IN WATER, SAMPLE 1 (7.8 pCi/l)

Lab	I-131 pCi/l ($\pm S_u$)[a]	$\bar{X}_{ij}$[b] (pCi/l)	S_{ij}[c] (pCi/l)	Accuracy[d] Index, A	Chemical Recovery (%)
10[f]	7.6 (0.67)				63.0
	7.7 (0.66)	7.5	0.30	96	70.0
	7.1 (0.66)				66.0
18[g]	6.7 (0.24)				90.9
	7.4 (0.28)	7.0	0.36	90	91.4
	7.0 (0.29)				94.5
19[h]	20.9 (3.96)				99.0
	23.0 (3.80)	23.1	2.28	297	99.0
	25.4 (3.62)				99.0

[a,b,c,d] See Table E-1 footnotes.

[f] Iodide isolated by anion exchange resin, purified by solvent extraction and assayed as palladium iodide by beta, gamma-coincidence counting.

[g] Method similar to f but assayed as cuprous iodide.

[h] Isolated by anion exchange resin which was gamma-counted with a sodium iodide detector.

TABLE F-2. RESULTS OF LABS USING INDEPENDENT METHODS FOR IODINE-131 IN WATER, SAMPLE 2 (25.9 pCi/l)

Lab	I-131 pCi/l (±S_u)[a]	$\bar{X}_{ij}$[b] (pCi/l)	S_{ij}[c] (pCi/l)	Accuracy[d] Index, A	Chemical Recovery (%)
10[f]	23.9 (1.53)				57.0
	22.6 (1.21)	23.0	0.72	89	68.0
	22.6 (1.18)				76.0
18[g]	23.6 (0.86)				93.3
	22.6 (0.82)	23.1	0.50	89	90.8
	22.9 (0.87)				93.3
19[h]	38.7 (6.98)				99.0
	31.3 (8.93)	41.1	11.22	159	99.0
	53.3 (9.78)				99.0

[a],[b],[c],[d]See Table E-1 footnotes.

[f],[g],[h]See Table F-1 footnotes.

TABLE F-3. RESULTS OF LABS USING INDEPENDENT METHODS FOR IODINE-131 IN WATER, SAMPLE 3 (78.3 pCi/l)

Lab	I-131 pCi/l ($\pm S_\mu$)[a]	$\bar{X}_{ij}$[b] (pCi/l)	S_{ij}[c] (pCi/l)	Accuracy[d] Index, A	Chemical Recovery (%)
10[f]	69.4 (2.51)				68.0
	63.3 (2.69)	66.6	3.13	85	69.0
	67.2 (2.79)				73.0
18[g]	70.1 (1.91)				91.5
	70.4 (1.03)	70.9	1.05	90	92.1
	72.1 (1.98)				88.8
19[h]	88.4 (10.58)				99.0
	98.8 (9.96)	91.0	7.6	116	99.0
	85.7 (10.12)				99.0

a,b,c,d See Table E-1 footnotes.

f,g,h See Table F-1 footnotes.